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POROSITY MEASUREMENT OF COMBUSTIBLE CARTRIDGE CASE MATERIALS

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FIRE CONTROL AND SMALL CALIBER
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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Research to develop an accurate and reproducible density test for 60-mm and 81-mm mortar propellant increment containers (combustible cartridge cases) was not successful. However, application of modern industrial porosity measurement techniques may afford superior and more applicable data than density obtained by displacement methods. Relationship to burning rate and strength is discussed. Further work to institute porosity as a specification is suggested.

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INTRODUCTION

Standard propellant densities are established by determining the weight of propellant occupying a known volume. Thus, two quantities must be known to determine density: weight and volume. Sample weight is relatively easy to measure, but the volume of the sample can pose significant problems depending on the sample's geometric and/or physical form. One example of this problem is evident in the case of granular propellant where the question arises: should the interstitial volume be a part of the unit volume in the density expression?

Bulk or apparent density is defined as weight per unit of "outside volume" (including intergranular voids). Volume of a granular solid can be determined by measuring the volume of liquid displaced by it, assuming that the liquid occupies all the interstices. This method is well documented and is commonly used to determine volumes, and thereby densitites, of a myriad of particulate solids. Choice of the displacement liquid has two practical restraints: (1) it must not dissolve the solid and (2) it must have a lower density than the solid. In actual practice, however, occluded air caused by excessive interfacial surface tension (between the liquid and solid being tested) can lead to erroneously high volume readings and correspondingly low densitites.

Porous solids with fibrous surface texture are especially prone to occlusion of air (bubbles), during density determinations. This phenomenon is precisely the crux in the unsuccessful programs for the development of a reproducible bulk density test for M204 (60-mm) and M205 (81-mm) mortar propellant increment containers (combustible cartridge cases).

This report indicates the inaccuracy and lack of precision in density determinations by conventional methods and introduces relatively new technology whereby porosity (void volume) is measured with accuracy far in excess of available density methods in such a manner that apparent (bulk) and true (skeletal, fiber) densities, pore size distribution with respect to to volume, and differential and integral surface areas can be calculated.

DENSITY

The need for an accurate, reproducible test method for density of M204/M205 propellant increment containers was recognized early in the development programs. The "nominal" density of 1.0, established by initial design research, indicated significant porosity (void volume) because all of the raw materials have a density higher than 1.0. The composite density calculation is given in table 1.

The density determination procedure recommended by Illinois Industrial and Technical Research Institute (IITRI) to Eldon Fiber Manufacturing Company (EFMC), the initial producer of combustible cartridge cases, was based on the Technical Association for Paper and Pulp Industry (TAPPI) method RC-256 (now called UM-18) (ref 1). The TAPPI method involved mercury displacement in a pycnometer which presented a problem for samples with the size and shape of the M205 propellant increment containers. The recommended method required modification of an analyt-

ical balance whereby the buoyant force of the submerged sample (upward force equivalent to the weight of mercury displaced) was measured. Reweighing the sample after immersion in mercury indicated no mercury had been absorbed. Unsuccessful efforts to simulate this technique encountered problems maintaining horizontal sample stability and duplicating the sensitivity (± 0.002) reported by IITRI.

This approach was abandoned in favor of using more traditional mercury displacement equipment and taking smaller "spot" samples.

The most useful instrument found was Amsler densitometer no. 9/601 (figs. 1 through 3) which measures sample volume by mercury displacement on a calibrated, advancing, precision-machined screw. Although visibility of the sample during the determination was impossible, the occlusion of air (as bubbles) was apparent from inspection of the density data. Initially three "spot samples" were cut from each of 14 different M205 propellant increment containers (from four different lots) and were weighed on an analytical balance. The samples were placed in the densitometer for volume determination. This data, given in table 2, indicated poor precision between containers from the same lot and samples from the same container; hence, no conclusions could be drawn about production uniformity with respect to density.

Further density development work was restricted to M205 containers produced by EFMC pulp molding process to minimize entrapment of air at the sample's surface (a major problem encountered with paper molding process samples which have "fuzzy surfaces"). In this series of analyses, only three M205 propellant increment containers were used. After each container was cut along the seam, circles of approximately 1/2 in. diameter were cut from each of the halves with a cork borer. Six circular samples were cut from each container and were weighed on an analytical balance. A minimum of six volume determinations (principal source of error) were made for each "spot" sample to ascertain the precision and accuracy of this method. These data, tabulated and summarized in table 3, indicate sensitivity at least on an order of magnitude lower than that claimed by IITRI.

POROSITY

Porosity is defined as the quantity of void spaces within a solid. Several methods of porosity measurement are proposed in the literature. One method which dealt with a comparison of bulk and true densities (equation 1) was not applicable in this study since a reproducible density test could not be developed.

$$\varepsilon = 1 - \frac{\rho_B}{\rho_T} \qquad (1)$$

ε = porosity
ρ_B = bulk density
ρ_T = true density

Another method which pressurizes mercury into the pores of the sample measures both the diameter and volume of the pores. It is generally known that in any system consisting of a porous solid and non-wetting liquid, there is a repulsion of the liquid from the surface of the solid. In 1921, E. W. Washburn (ref 2) proposed a method for measuring the pressure required to force mercury into the pores of an evacuated porous material, and related this pressure to the radius of the pores thus penetrated (equation 2):

$$P = \frac{2 \gamma \cos \theta}{r} \qquad (2)$$

P = pressure
γ = surface tension of the liquid
θ = contact angle (solid/liquid)
r = radius of smallest pore penetrated at pressure, P

In the Washburn procedure, a dry granular solid was weighed, placed in a steel pressure bomb, and evacuated to remove adsorbed gases. The bomb was then filled with mercury, and a series of pressure and volume measurements were made as the pressure was increased stepwise. Any decrease in volume (ΔV) accompanying a pressure increase (ΔP) is due to penetration of pores of effective radii between r and r $-\Delta$ r

$$\frac{\Delta r}{\Delta P} = \frac{-2 \gamma \cos \theta}{P^2} \qquad (3)$$

Thus, if various pore sizes are present, the volume fraction of total porosity consisting of pores between any two stated diameters can be determined. A "blank" experiment without porous material was suggested to determine the correction for compressibility of mercury and expansion of the bomb under pressure.

Further improvements reported by Winslow and Shapiro (ref 3) led to construction of the prototype porosimeter shown in figure 4. This instrument extended the range of pore size determination by including pores much less than 1 micron diameter as well as many microns in diameter.

Pore analysis on the prototype porosimeter typically followed this procedure:

1. A sample of suitable size [total contained pore volume within the calibrated limits of the penetrometer tube (E)] was placed within the ground glass joint (G).

2. The assembled penetrometer (E and G) was placed within a filling device which was then evacuated.

3. Mercury was introduced to fill the penetrometer while it was still under vacuum.

4. Admission of atmospheric pressure caused the penetrometer to fill, immersing the sample without exposure to air.

5. The filled penetrometer was then transferred from the filling device to the porosimeter (fig. 4).

6. Pressure was applied in a stepwise manner up to about 600 psi. At each selected pressure level, the mercury level was read on the calibrated penetrometer stem. The volume of pores in a given size interval would be the difference from the previous reading.

No corrections for distortion were required since the penetrometer was subjected internally and externally to the same pressure. The entire procedure required about 20 minutes.

Penetrometer design allows for weighing of the filled assembly on an analytical balance. This data, in conjunction with sample and empty penetrometer weight, provides a basis for calculation of apparent density. Calculation of pore volumes not in the range of the instrument (> 10 microns, < 0.3 micron) is also possible if real and apparent densities are known.

In their paper, Winslow and Shapiro (ref 3) also discussed preparation of a reference standard. This standard was a block of nickel 0.948 cm long, 0.838 cm wide and 0.588 cm high. The block was drilled 70 times (top to bottom) with U.S. standard drill no. 76 (508 μ dia). These "pores" varied from the diameter of the drill and from true cylindrical shape only within the limits of accuracy and perfection of the drilling. The actual pore diameter was approximated in two ways:

First, microscopic measurement of the pore mouths indicated a variance of 511μ to 618μ on the "drill-in" side (average = 534μ) and 509μ to 753μ on the "drill-out" side (average 545μ). From both sides the pores appeared cylindrical except for shallow skin fractures where the drill emerged from the metal.

A second approximation of the true pore diameter was calculated from the weight and external dimensions of the drilled block and the real density of nickel. Assuming that the length of each pore was 0.588 cm, the calculated pore diameter is 561μ. Thus, an average "pore" diameter of 550μ can be concluded on the basis of these approximations. Mercury intrusion data are compared with the aforementioned approximations in table 4.

Originally, cylindrically shaped pores were assumed in calculation of surface areas from mercury intrusion porosimetry data. However, current theories allow these calculations directly from porosimeter pressure/volume curves without assuming any particular pore geometry (ref 4). Areas thus calculated compare favorably with those measured by BET nitrogen desorption (table 5).

The proposed equation for surface area is given (equation 4):

$$A = \frac{-1}{m \cos \theta \gamma_L} \int_o^{V \max} P dV \qquad (4)$$

where: A = area per gram sample
m = mass of sample used in porosimeter

Θ = contact angle
γ_L = surface tension porosimeter liquid
V_{max} = apparent total pore volume
P = measured porosimeter pressure
dV = incremental volume instrusions

If the porosimeter measurements have been made with mercury at 25°C, γL = 480 dynes/cm and Θ is assumed to be 130°, the equation becomes

$$A = \frac{0.02253}{m} \int_o^{V_{max}} P dV \qquad (5)$$

The only assumptions which were made in this derivation were those which applied to mercury intrusion porosimetry: mercury does not wet the sample, porosimeter pressures are high enough for mercury to penetrate the smallest pores, and no "ink-bottle" pores (where the opening is smaller than the largest inside diameter) are present.

Three M205 (81-mm mortar) propellant increment containers were sent to American Instrument Company (AMINCO) on 4 December 1979 to ascertain any measurable differences in pore structure which could serve as a basis for development of a suitable bulk density test. One of the samples sent was made by EFMC via a pulp molding process; the other two represented the "range of production" (hard and soft) by Lory Industries' paper molding process. These samples had previously been tested at ARRADCOM for hardness (Shore "A" Durometer) and compression (MIL-C-48882B with Amendment 2). These values along with the total porosity measured at 60,000 psi by AMINCO are given in table 8.

The porosimetry results reported by AMINCO on 14 January 1980 included porosity and density determination data sheets (summarized in table 7) and a pore size distribution curve for each sample (appendix A). The Lory "hard" sample was rerun because the lower porosity required a larger sample. Aside from the much lower porosity measured in the "hard" sample, both samples from Lory Industries exhibited a broader distribution of pore diameters than the EFMC sample. The density reported in table 7 refers to a true (fiber) density excluding pore volume. These absolute densities are roughly equivalent (1.82, 1.77, 1.79 g/cm^3), as expected, since the compositions are chemically identical.

A porosity analysis of another EFMC propellant increment container was provided by Porous Materials Inc. (PMI) in September 1980 (table 8).

This initial analysis by PMI was followed up by a visit to their facility by ARRADCOM personnel in November 1980. At that time, eleven automated porosity determinations (by mercury intrusion) on nine mortar propellant increment containers were observed. Each container had previously been tested for strength (compression) and permeability ("back pressure" when subjected to 5 psig internal air pressure). These data are compared to the measured porosity in table 9. Three of the samples were M204 containers and six were M205 containers. Three of the M205 containers were made by EFMC and the other M205's were Lory products. One of the EFMC containers was cut up and samples for porosity were taken from the top, bottom, and ends to determine homogeneity. From the data it appears

that strength (as measured by compression) is inversely proportional to porosity. The relationship between porosity and permeability, however, is not quite so obvious since only the Lory hard M204 (80A002-003) had an unusually high "back pressure." Lack of precision between samples from the same EFMC container indicate a surprising lack of homogeneity within the sample analyzed.

In November 1980, Quantachrome, another supplier of porosimeters, provided an analysis of samples of Lory's "kidney-cut" NC paper and of a molded M204 half-container. Results of this analysis are given below:

	Sample weight (g)	Maximum pressure (psi)	Measured porosity (cm^3/g)	Surface area (m^2/g)
"Kidney-cut" paper	0.2988	15	1.422	
"Kidney-cut" paper	0.1246	300	1.966	2.5
Molded M204 half-containers	0.3212	300	0.592	

These results indicate that the porosity of the molded container is approximately one-third the porosity of the paper stock used. This finding is logical since the stock paper thickness (0.045 in. to 0.055 in.) is about three times that of the molded container (0.014 in. to 0.022 in.). The paper sample had to be run twice because its porosity exceeded the capacity of the penetrometer stem for the sample size used. Data interpretations offered by Quantachrome maintained that porosity measurement at 60,000 psi was unnecessary because most of the pores were intruded at 300 psi (indicating pore diameters > 0.6 microns).

The only other known supplier of mercury intrusion porosimeters, Micromeritics Instrument Corporation, was contacted regarding an analysis of an M205 container made by EFMC. Results showed that the measured porosity (0.456 cm^3/g) was in close agreement with values determined by the other porosimeter suppliers for similar EFMC samples. A pore-size distribution curve and a density determination sheet are included in appendix B.

Names and addresses of all U.S. porosimeter suppliers are listed in appendix C.

EFMC and Lory "hard" M205 propellant increment containers were analyzed by Energetics Materials Division, LCWSL, ARRADCOM, for BET surface area. The results were:

	BET surface area (m^2/g)
EFMC	1.169
Lory "hard"	1.478

The values are in agreement with values calculated from mercury intrusion porosimetry data (equation 5).

CONCLUSIONS

An accurate and reproducible density test for the M204 and M205 propellant increment containers was not developed. However, measurement of porosity by mercury intrusion porosimetry can suffice as an alternate, perhaps superior, production control parameter. Additional analyses will be required to provide a sufficient data base for production specification. It is known that Lory Industries' "hard" containers burn slower and less completely than EFMC or Lory "soft" containers; therefore, the data may indicate that the burning rate is directly proportional to porosity for a given container composition. Although maximum burning rate is desirous, porosity must be compromised with physical strength since a very porous container (for example, Lory "soft") fails sequential rough handling tests (TOP 4-2-602). From the data in tables 6 and 9 it can be concluded that strength is inversely proportional to porosity for a given container composition. Therefore, optimum porosity can only be established with respect to both burning rate and physical strength.

RECOMMENDATIONS

1. A program should be conducted to determine the optimum porosity of M204 and M205 propellant increment containers with respect to:

   a. buring rate and/or gun performance

   b. strength.

2. Porosity, as measured by mercury intrusion porosimetry, should be instituted as a production specification in lieu of "nominal density" for M204 and M205 propellant increment containers.

3. Application of porosimetry to other combustible case designs should be explored.

4. Efficacy of a computer prediction of burning rate and gun performance of combustible cartridge cases, based on incremental porosimetry date, should be determined.

REFERENCES

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2. E. W. Washburn, "Note on a Method of Determining the Distribution of Pore Sizes in a Porous Material," Proceedings, National Academy Science, vol 7, 1921, page 115.

3. N. M. Winslow and J. J. Shapiro, "An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration," ASTM Bulletin, February 1959, page 39.

4. H. M. Rootare and C. F. Prenzlow, "Surface Areas from Mercury Porosimeter Measurements," Journal of Physical Chemistry, vol 71, 1967, page 2733.

Table 1. Composite density calculation

	Density (g/cm^3)	Density (cm^3/g)	Weight Fraction	Product
Nitrocellulose (NC)	1.66	0.6024	0.78	0.469879
Diphenylamine (DPA)	1.159	0.8628	0.01	0.008628
Resin (polyvinylactate)	1.19	0.8403	0.07	0.058823
Kraft fibers (cellulose)	1.58	0.6329	0.0375	0.023734
Acrylic fibers (polyacrylonitrile)	1.175	0.8511	0.0375	0.031914
Polyester fibers (PE terephthalate)	1.39	0.7194	0.065	0.046762
			1.0000	0.639740

Composite density $= \frac{1}{0.63974} = 1.5631\ g/cm^3$.

Table 2. M205 (81-mm mortar) propellant increment container densities measured by mercury displacement

Production data	Weight (g)	Volume (cm^3)	Density (g/cm^3)	Weight (g)	Volume (cm^3)	Density (g/cm^3)	Weight (g)	Volume (cm^3)	Density (g/cm^3)	Average Density (g/cm^3)	Standard deviation
Lory "double-dipped"	0.0574	0.072	0.797	0.0541	0.069	0.784	0.0545	0.069	0.790	0.790	0.007
	0.0471	0.066	0.714	0.0461	0.072	0.640	0.0545	0.078	0.699	0.684	0.039
	0.0397	0.075	0.529	0.0415	0.057	0.728	0.0392	0.069	0.568	0.608	0.106
	0.0625	0.078	0.801	0.1072	0.186	0.576	0.1213	0.189	0.642	0.673	0.116
								Group avg		0.689	0.097
EFMC	0.0431	0.048	0.898	0.0441	0.063	0.700	0.0427	0.051	0.837	0.812	0.101
	0.0433	0.054	0.802	0.0444	0.054	0.822	0.0455	0.066	0.689	0.771	0.072
	0.0507	0.054	0.939	0.0489	0.063	0.776	0.0364	0.060	0.607	0.774	0.166
	0.0475	0.063	0.754	0.0501	0.048	1.044	0.0488	0.048	1.017	0.938	0.160
								Group avg		0.824	0.132
Lory "soft"	0.0310	0.060	0.517	0.0328	0.078	0.421	0.0293	0.087	0.337	0.425	0.090
	0.0825	0.108	0.764	0.1031	0.150	0.687	0.0611	0.099	0.617	0.689	0.074
	0.1435	0.180	0.797	0.1395	0.153	0.912	0.1517	0.168	0.903	0.871	0.064
	0.0966	0.189	0.511	0.0820	0.138	0.594	0.1134	0.153	0.741	0.615	0.117
								Group avg		0.650	0.183
Lory "hard"	0.0517	0.063	0.821	0.0485	0.057	0.851	0.0490	0.060	0.817	0.830	0.019
	0.1589	0.123	1.292	0.1226	0.099	1.238	0.1344	0.120	1.120	1.217	0.088
								Group avg		1.023	0.220

Table 3. Density test results for M205 container assemblies

	SAMPLE NO.
REPORT FROM THE ENERGETIC MATERIALS DIVISION, Chem. Br., Analtyical Sect	DATE 30 Jun 80
KIND OF SAMPLE Felted Increment, 81-mm	REPORT NO. EMD MD 31 80
RECEIVED FROM Propulsion Tech. Branch, Applied Science Div., LCWSL	DATE RECEIVED
REFERENCE OF X. O. 1900-01-003 Engineer: Mr. C. Manning	
REPRESENTING 81-mm Mortar Increments from EFMC Corporation	

Summary of Density, (g/cm^3), Values for 81-mm Increment Containers

Increment No	1		3		4	
Specimen Location	Av. Density	Std. Dev	Av. Density	Std. Dev.	Av Density	Std Dev
Top End	0.937	0.017	0.924	0.037	0.938	0.033
Top Side	0.961	0.039	0.885	0.025	0.920	0.040
Top Middle	0.933	0.025	0.918	0.013	0.918	0.020
Bottom End	0.591	0.011	0.854	0.034	0.802	0.019
Bottom Side	0.668	0.020	0.843	0.035	0.805	0.035
Bottom Middle	0.672	0.019	0.920	0.015	0.839	0.016

Individual Values for Volume, cm^3 and Density, g/cm^3 for 81-mm Increment Container

Increment No.	1		3		4	
Specimen Location	Top End		Top End		Top End	
Specimen Wt, gms	0.0780		0.0778		0.0736	
Determination No.	Volume, (cm^3)	Density, (g/cm^3)	Volume, (cm^3)	Density, (g/cm^3)	Volume, (cm^3)	Density, (g/cm^3)
1	0.0834	0.935	0.0864	0.900	0.0789	0.933
2	0.0837	0.932	0.0873	0.891	0.0738	0.997
3	0.0846	0.922	0.0783	0.994	0.0774	0.951
4	0.0846	0.922	0.0855	0.910	0.0804	0.915
5	0.0837	0.932	0.0843	0.923	0.0792	0.929
6	0.0804	0.970	0.0840	0.926	0.0813	0.905
7	0.0822	0.949	-	-	-	-
Avg.		0.937	Avg.	0.924	Avg.	0.938
Std.Dev.		0.017	Std.Dev.	0.037	Std.Dev	0.033

Table 3. (cont)

Increment No.	1		3		4	
Specimen Location	Top Side		Top Side		Top Side	
Specimen Wt, gms	0.0730		0.0745		0.0722	
Determination No.	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)
1	0.0801	0.911	0.0870	0.856	0.0789	0.915
2	0.0774	0.943	0.0801	0.931	0.0783	0.922
3	0.0729	1.001	0.0837	0.890	0.0816	0.885
4	0.0729	1.001	0.0855	0.871	0.0819	0.882
5.	0.0768	0.951	0.0846	0.881	0.0726	0.994
6	-	-	0.0846	0.881	0.0783	0.922
	Avg.	0.961	Avg.	0.885	Avg.	0.920
	Std.Dev.	0.039	Std.Dev.	0.025	Std.Dev.	0.016

Increment No.	1		3		4	
Specimen Location	Top Middle		Top Middle		Top Middle	
Specimen Wt, gms	0.0747		0.0748		0.0747	
Determination No.	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)
1	0.0831	0.899	0.0807	0.927	0.0798	0.936
2	0.0813	0.919	0.0834	0.900	0.0828	0.902
3	0.0771	0.969	0.0813	0.920	0.0831	0.900
4	0.0783	0.954	0.0819	0.913	0.0795	0.940
5	0.0804	0.930	0.0798	0.937	0.0819	0.912
6	0.0807	0.926	0.0822	0.910	0.0813	0.919
	Avg.	0.933	Avg.	0.918	Avg.	0.918
	Std.Dev.	0.025	Std. Dev.	0.013	Std.Dev.	0.020

Increment No.	1		3		4	
Specimen Location	Bottom End		Bottom End		Bottom End	
Specimen Wt, gms	0.0555	0.	0.0747		0.0712	
Determination No.	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)
1	0.0969	0.573	0.0873	0.856	0.0933	0.763
2	0.0933	0.595	0.0897	0.833	0.0894	0.796
3.	0.0948	0.585	0.0897	0.833	0.0873	0.816
4	0.0930	0.597	0.0849	0.880	0.0867	0.821
5	0.0942	0.589	0.0918	0.814	0.0861	0.827
6	0.0918	0.605	0.0825	0.905	0.0900	0.791
	Avg.	0.591	Avg.	0.854	Avg.	0.802
	Std.Dev.	0.011	Std.Dev.	0.034	Std.Dev.	0.019

Table 3. (cont)

Increment No	1		3		4	
Specimen Location	Bottom Side		Bottom Side		Bottom Side	
Specimen wt, gms	0.0597		0.0738		0.0688	
Determination No.	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g)
1	0.0915	0.652	0.0861	0.857	0.0903	0.762
2	0.0876	0.682	0.0873	0.845	0.0804	0.856
3	0.0933	0.640	0.0891	0.828	0.0870	0.791
4	0.0861	0.693	0.0897	0.823	0.0870	0.791
5	0.0894	0.668	0.0852	0.866	0.0819	0.840
6	0.0885	0.675	0.0882	0.837	0.0870	0.791
	Avg.	0.668	Avg.	0.843	Avg.	0.805
	Std.Dev.	0.020	Std.Dev.	0.035	Std.Dev.	0.035

Increment No	1		3		4	
Specimen Location	Bottom Middle		Bottom Middle		Bottom Middle	
Specimen wt, gms	0.0585		0.0786		0.0697	
Determination No.	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g/cm^3)	Volume (cm^3)	Density (g)
1	0.0876	0.668	0.0876	0.897	0.0807	0.864
2	0.0861	0.679	0.0837	0.939	0.0846	0.824
3	0.0843	0.694	0.0846	0.929	0.0843	0.827
4	0.0915	0.639	0.0858	0.916	0.0831	0.839
5	0.0876	0.668	0.0864	0.910	0.0840	0.830
6	0.0855	0.684	0.0846	0.929	0.0819	0.851
	Avg.	0.672	Avg.	0.920	Avg.	0.839
	Std.Dev.	0.019	Std.Dev.	0.015	Std.Dev.	0.016

Method: Mercury displacement with the Amsler 9/601 instrument.

Table 4. Comparison of porosimetry reference standard porosities (calculated and measured)

		Calculated	Measured
Volumes (cm^3)	Apparent	0.554	0.551
	Pore	0.108	0.105[a]
			0.104[b]
	Real	0.446	0.446[a]
			0.447[b]
Densities (g/cm^3)	Apparent	7.16	7.20
	Real	8.90	8.88

[a] First determination

[b] Second determination

Table 5. Comparison of surface areas calculated from mercury intrusion data with BET measurements

Sample	Calculated area (m^2/g)	BET measured area (m^2/g)
Aluminum dust	1.35	1.25
Anatase (TiO_2)	15.1	10.3
Boron nitride	19.6	20.0
Calcium cyanamide	2.75	3.17
Spheron -6 (carbon black)	107.8	110.0
Sterling FT (carbon)	15.7	12.3
Powdered copper	0.34	0.49
Fluorospan	2.48	2.12
Fly ash	2.34	2.06
Borosilicate alkaliglass (porous)	11.0	7.9
Hydroxylapatite	55.2	55.0
Iron oxide	14.3	13.3
Powdered iron	0.20	0.30
Silver iodide	0.48	0.53
Powdered tungsten	0.11	0.10
Tungsten carbide	0.11	0.14
Vanadium oxide catalyst	0.40	0.40
Zinc dust	0.34	0.32
Zinc powder	1.47	1.60
Zinc powder	2.16	2.00

Table 6. Comparison of porosity to strength

	M205 container		
	Lory "soft"	EFMC	Lory "hard"
Porosity* (cm^3/g)	0.586	0.474	0.218
Density* (g/cm^3)	0.8737	0.9553	1.268
Compression (lb)	1.85	5.40	4.00
Hardness (durometer)	35	70	55

* Calculated from AMINCO porosimetry data.

Table 7. Summary of AMINCO density data

	M205 container assembly			
	Lory "soft"	EFMC	Lory "hard"	Lory "hard" (rerun)
Penetrometer volume (cm^3)	5.6009	5.6009	5.6009	5.6009
Weight penetrometer, sample and mercury (g)	142.5838	142.4928	142.3437	139.5765
Weight of penetrometer and sample (g)	68.7238	68.7676	67.9147	68.1582
Weight of mercury (g)	73.8600	73.7252	74.4290	71.4183
Volume of mercury at 26°C (cm^3)	5.4584	5.4464	5.4994	5.2760
Volume of sample plus pores (cm^3)	0.1425	0.1545	0.1015	0.3249
Sample weight (g)	0.1245	0.1476	0.1287	0.4271
Bulk density (g/cm^3)	0.8737	0.9553	1.268	1.315
Mercury penetration at 60,000 psi (cm^3)	0.074	0.071	0.029	0.074
Volume of sample - pore volume (cm^3)	0.0685	0.0835	0.0725	0.2509
True density (g/cm^3)	1.82	1.77	1.79	1.70

Table 8. Complimentary pore analysis by Porous Materials Inc.*

Pressure (psi)	Cumulative penetration volume (cm^3/g)
0.91	0.0072
1.68	0.0216
3.58	0.0435
6.22	0.0664
9.40	0.0898
12.30	0.1120
15.00	0.1301
18.13	0.1607
20.16	0.1900
23.06	0.2295
27.15	0.2751
31.56	0.3145
37.77	0.3507
50.99	0.3819
72.62	0.4055
155.55	0.4287
399.48	0.4396
599	0.4428
999	0.4475
1499	0.4507
3000	0.4514
5000	0.4514
7500	0.4539

* Sample = EFMC M205 container
Porosimetry sample weight = 0.4200 g

Table 9. Comparison of porosity[a] to back pressure and compression data

Container	MFR	Lot	Back pressure (psig)	Compression (lb)	Porosity (cm^3/g)	Surface area[b] (m^2/g)
M204	Brunswick	2-1	6.6	4.80	0.4483	1.403
M204	Lory	80A003-001	6.3	1.67	0.7560	1.410
M204	Lory	80A002-003	11.5	5.40	0.2276	3.664
M205	Lory	"soft"	5.1	1.08	1.2950	-
M205	Lory	"hard"	5.6	3.20	0.3482	-
M205	Lory	"double-dipped"	5.2	2.43	0.7724	-
		"A" - (top)	5.7	4.80	0.2969	-
M205	EFMC	"A" - (bottom)	5.7	4.80	0.6634	-
		"A" - (ends)	5.7	4.80	0.4076	-
M205	EFMC	"B"	5.7	4.17	0.5823	-
M205	EFMC	"C"	5.9	5.02	0.4965	-

[a] Determined by PMI.

[b] Calculated from mercury intrusion data (equation 3).

Figure 1. Amsler densitometer no. 9/601

Figure 2. Amsler densitometer (disassembled)

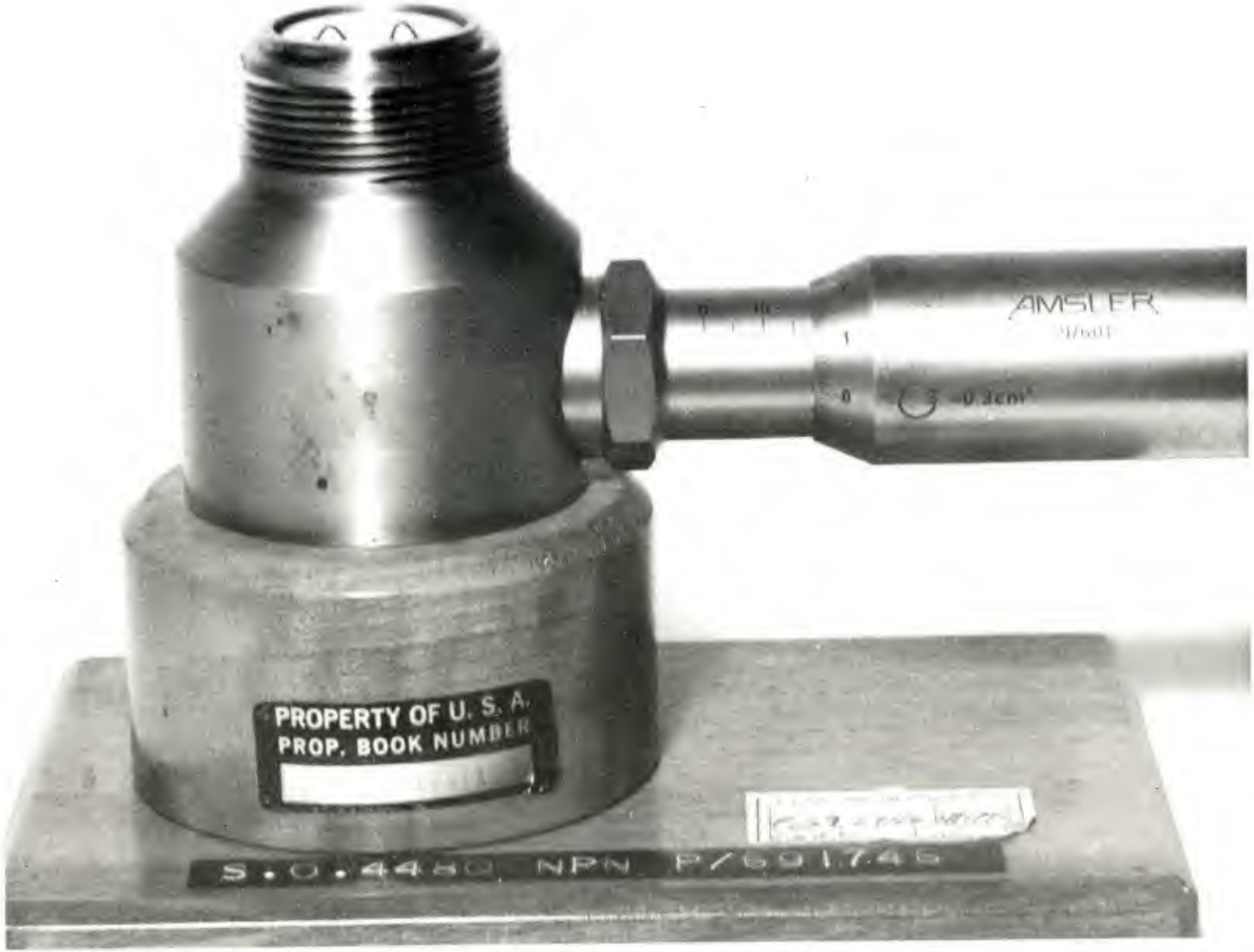
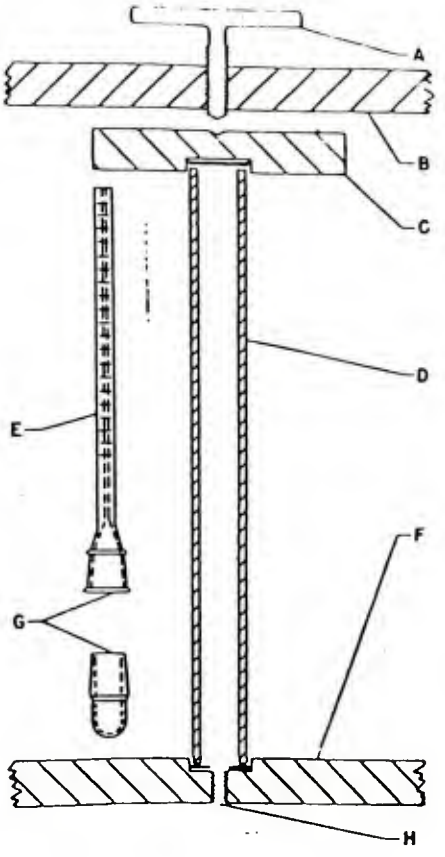


Figure 3. Amsler densitometer (close-up showing calibrations)



(A) Hand screw

(B) Yoke

(C) Upper closure

(D) Glass pressure tube

(E) Calibrated apillary, 0.2 ml capacity, 0.002 ml intervals

(F) Lower closure

(G) Glass joint, 19/22 standard taper

(H) Threaded port for connection with pressure sources, gages, and vent.

Figure 4. Diagram of prototype porosimeter and penetrometer

APPENDIX A

AMINCO PORE SIZE DISTRIBUTION CURVES

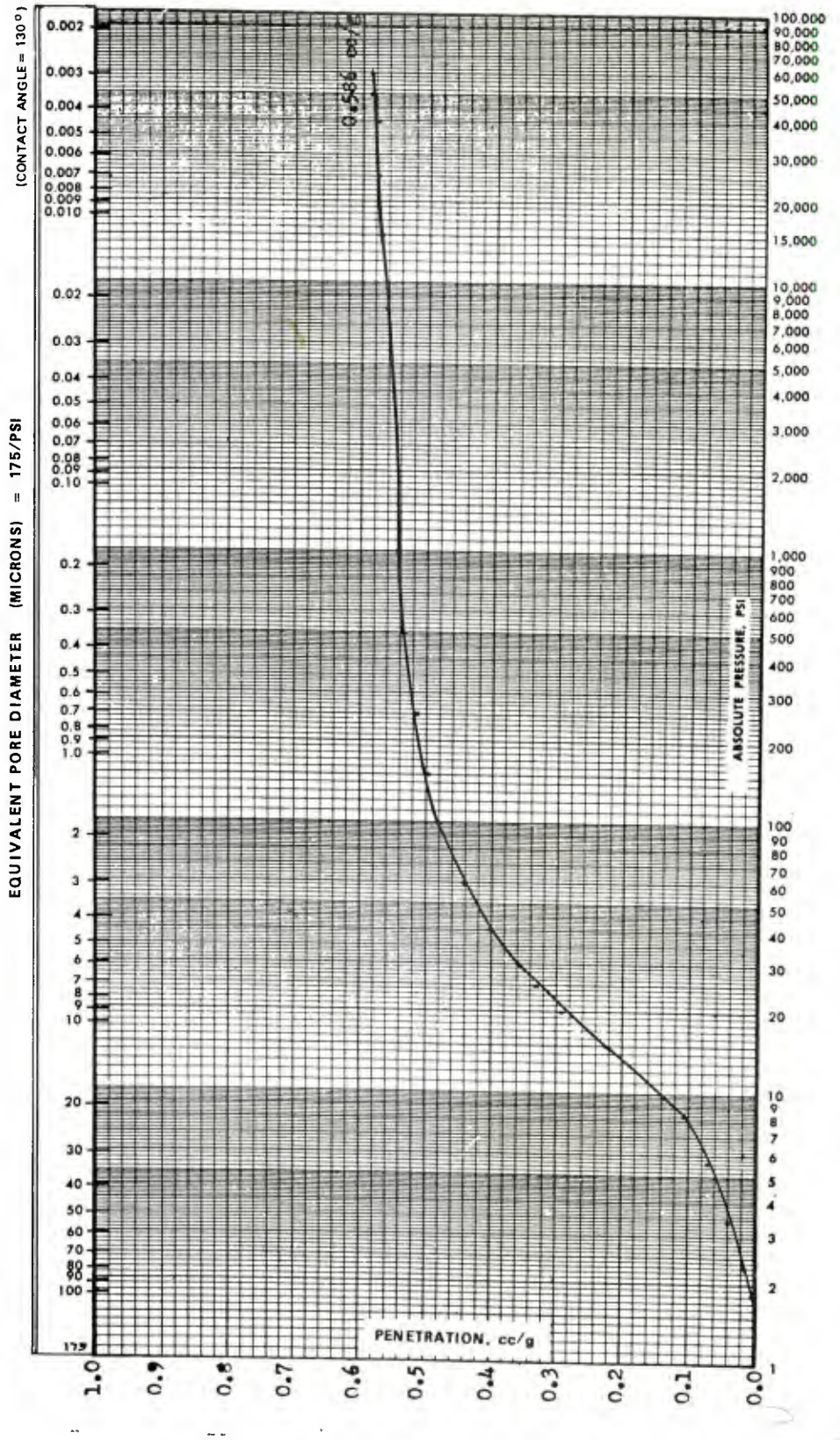


Figure A-1. Sample M205 container assembly "Lory soft" (paper molding process) porosimeter sample weight 0.1245 g

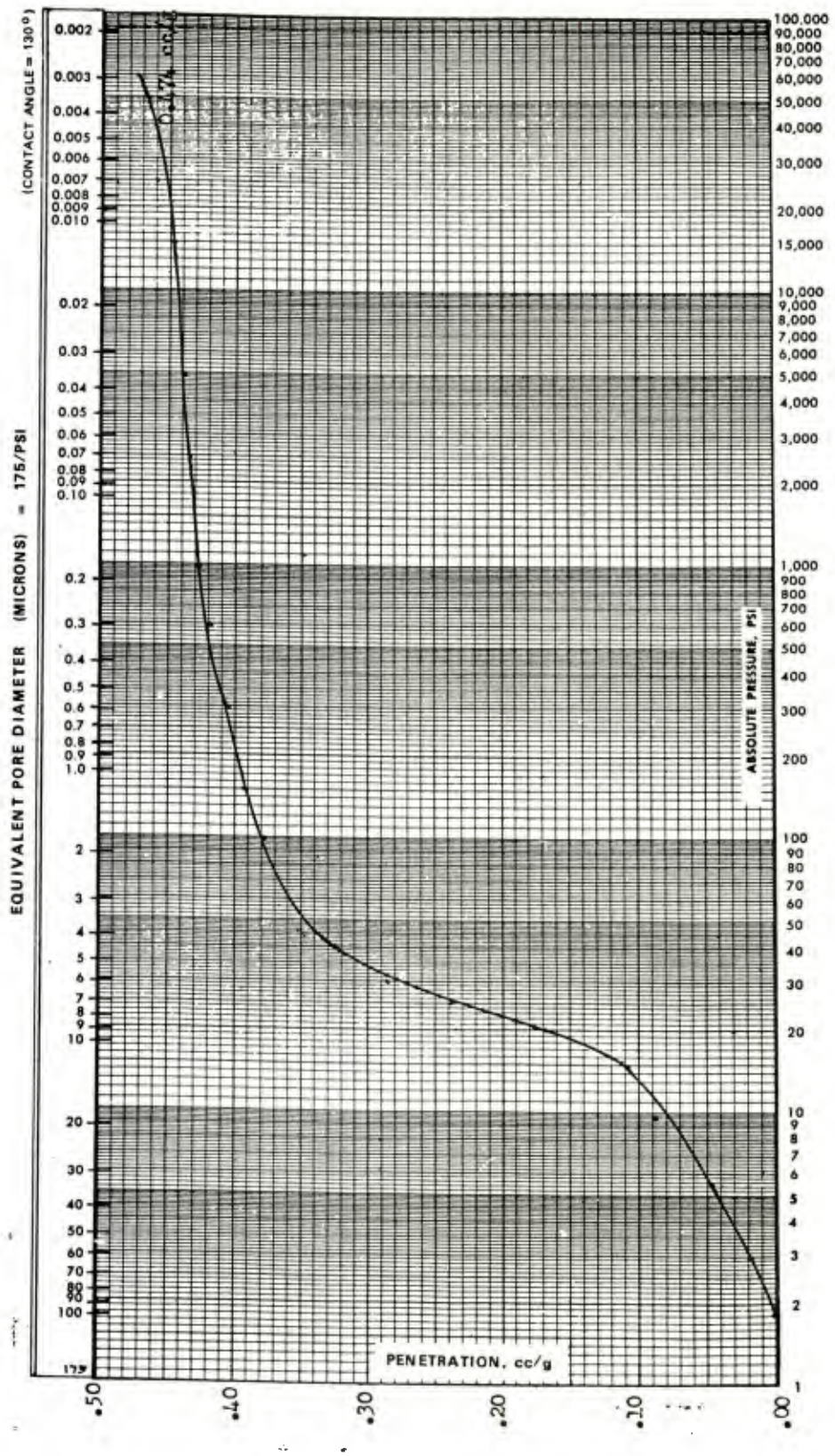


Figure A-2. Sample M205 container assembly "EFMC" (pulp molding process) porosimeter sample weight 0.1476 g

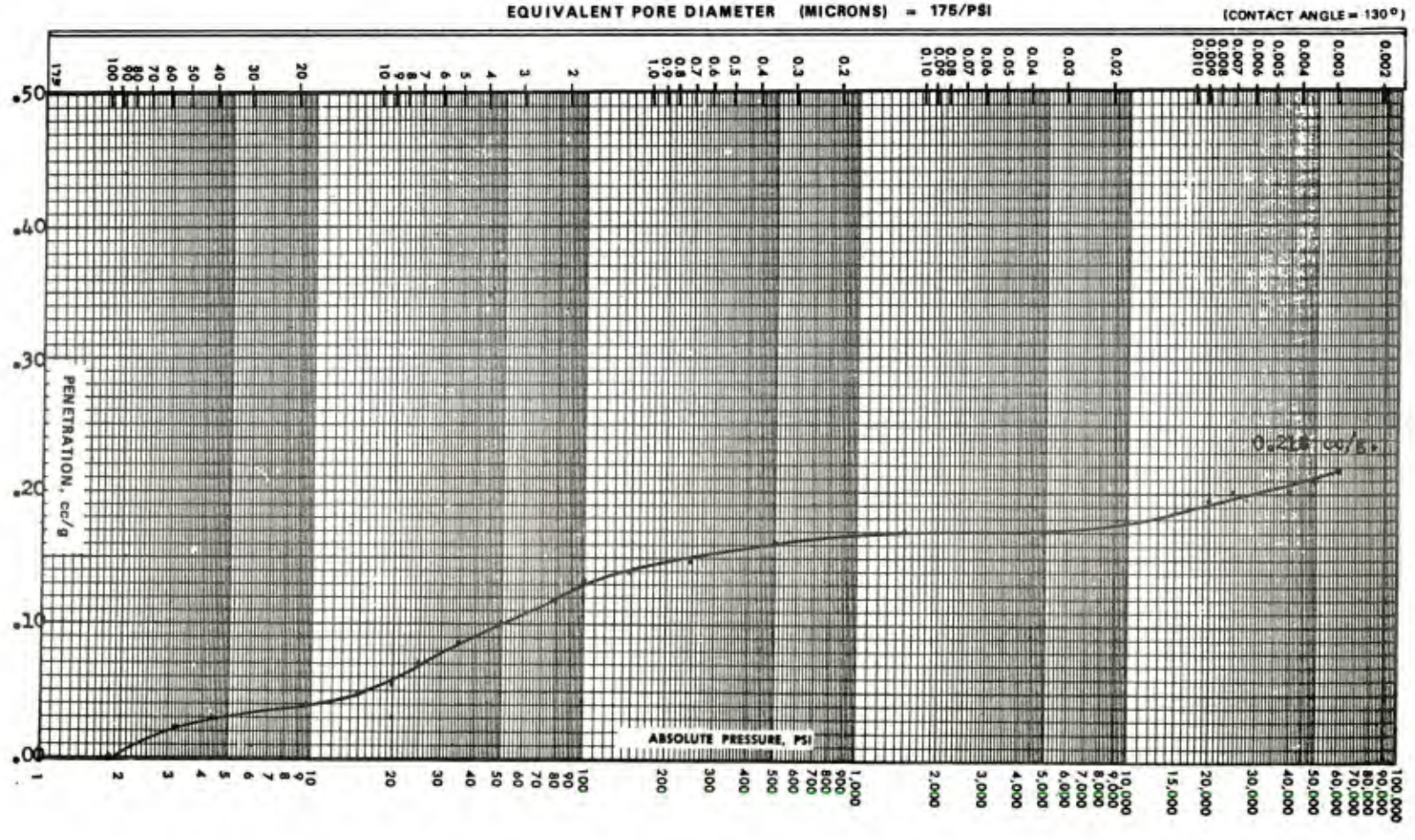


Figure A-3. Sample M205 container assembly "Lory hard" (paper molding process) porosimeter sample weight 0.1287 g

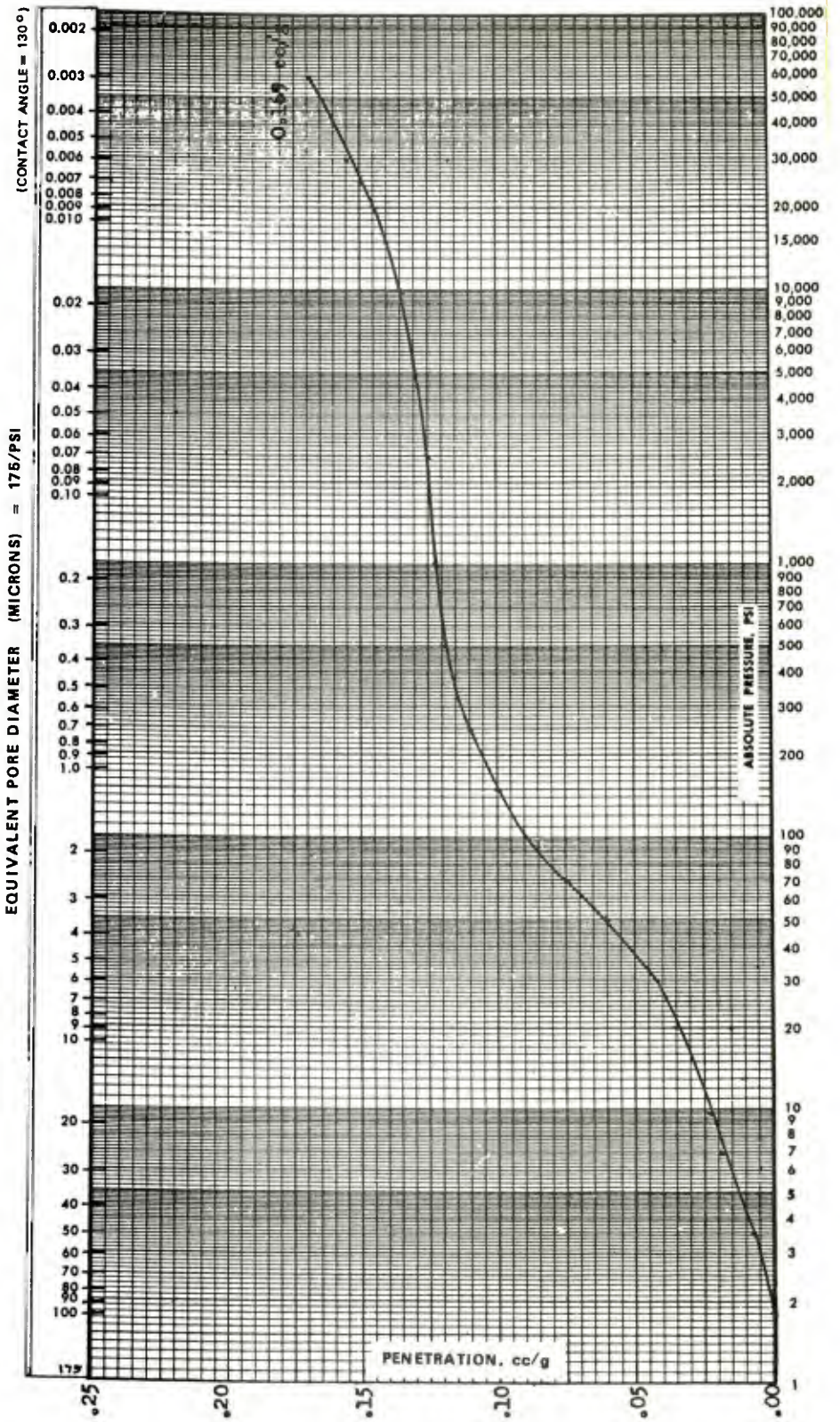


Figure A-4. Sample M205 container assembly "Lory soft" (paper molding process) porosimeter sample weight (rerun) 0.4271 g

APPENDIX B

MICROMERITICS PORE SIZE DISTRIBUTION CURVE

DENSITY DETERMINATION BY MERCURY POROSIMETRY

DENSITY DETERMINATION BY MERCURY POROSIMETRY

SAMPLE ARRADSORB PICRTINNY NITROCELLULOSE PAPER DATE 1/13/81

BY

A. Calibration of penetrometer volume: #330

1. Weight of penetrometer filled with mercury . . . . . g
2. Weight of sealed, empty penetrometer . . . . . g
3. Weight of mercury (1 - 2)* . . . . . g

Room temperature ____ °C; Density of mercury** 13.5 g/cc

4. Volume of penetrometer (3 ÷ density of mercury) . . . . . 6.2049 cc

B. Bulk density calculation:

5. Weight of sample (7 - 2) . . . . . 0.5252 g
6. Weight of penetrometer, sample & mercury . . . . . 149.923 g
7. Weight of penetrometer & sample . . . . . 73.552 g
8. Weight of mercury (6 - 7) . . . . . 76.371 g

Room temperature 23.6 °C; Density of mercury** 13.5374 g/cc

9. Volume of mercury (8 ÷ density of mercury) . . . . . 5.6415 cc
10. Volume of sample (4 - 9) . . . . . 0.5634 cc
11. Bulk volume of sample (10 ÷ 5) . . . . . 1.0727 cc/g*
12. Bulk density of sample (reciprocal of 11) . . . . . 0.9322 g/cc

C. Apparent density calculation:

13. Pore volume, mercury displaced in penetrometer stem . . . 0.2395 cc

at a maximum pressure of . . . . . 32 000 psia

14. Volume of sample, less pore volume (10 - 13) . . . . . 0.3239 cc
15. Specific volume of sample (14 ÷ 5) . . . . . 0.6167 cc/g
16. Apparent density of sample (reciprocal of 15) . . . . . 1.6215 g/cc

*NOTE: *Numbers in parentheses indicate quantity entered in the blank of the line designated by the number.*

DENSITY OF MERCURY**

°C	g/cc						
18.0	13.5512	23.2	13.5384	25.2	13.5335	27.2	13.5286
19.0	13.5487	23.4	13.5379	25.4	13.5330	27.4	13.5281
20.0	13.5462	23.6	13.5374	25.6	13.5325	27.6	13.5276
21.0	13.5438	23.8	13.5369	25.8	13.5320	27.8	13.5271
22.0	13.5413	24.0	13.5364	26.0	13.5315	28.0	13.5266
22.2	13.5408	24.2	13.5359	26.2	13.5310	29.0	13.5242
22.4	13.5403	24.4	13.5354	26.4	13.5305	30.0	13.5217
22.6	13.5399	24.6	13.5350	26.6	13.5301	31.0	13.5193
22.8	13.5394	24.8	13.5345	26.8	13.5296	32.0	13.5168
23.0	13.5389	25.0	13.5340	27.0	13.5291	33.0	13.5144

COMMENTS: SAMPLE WAS EVACUATED DOWN TO 7 μm
61% OF HG IN PENETROMETER STEM WAS DISPLACED

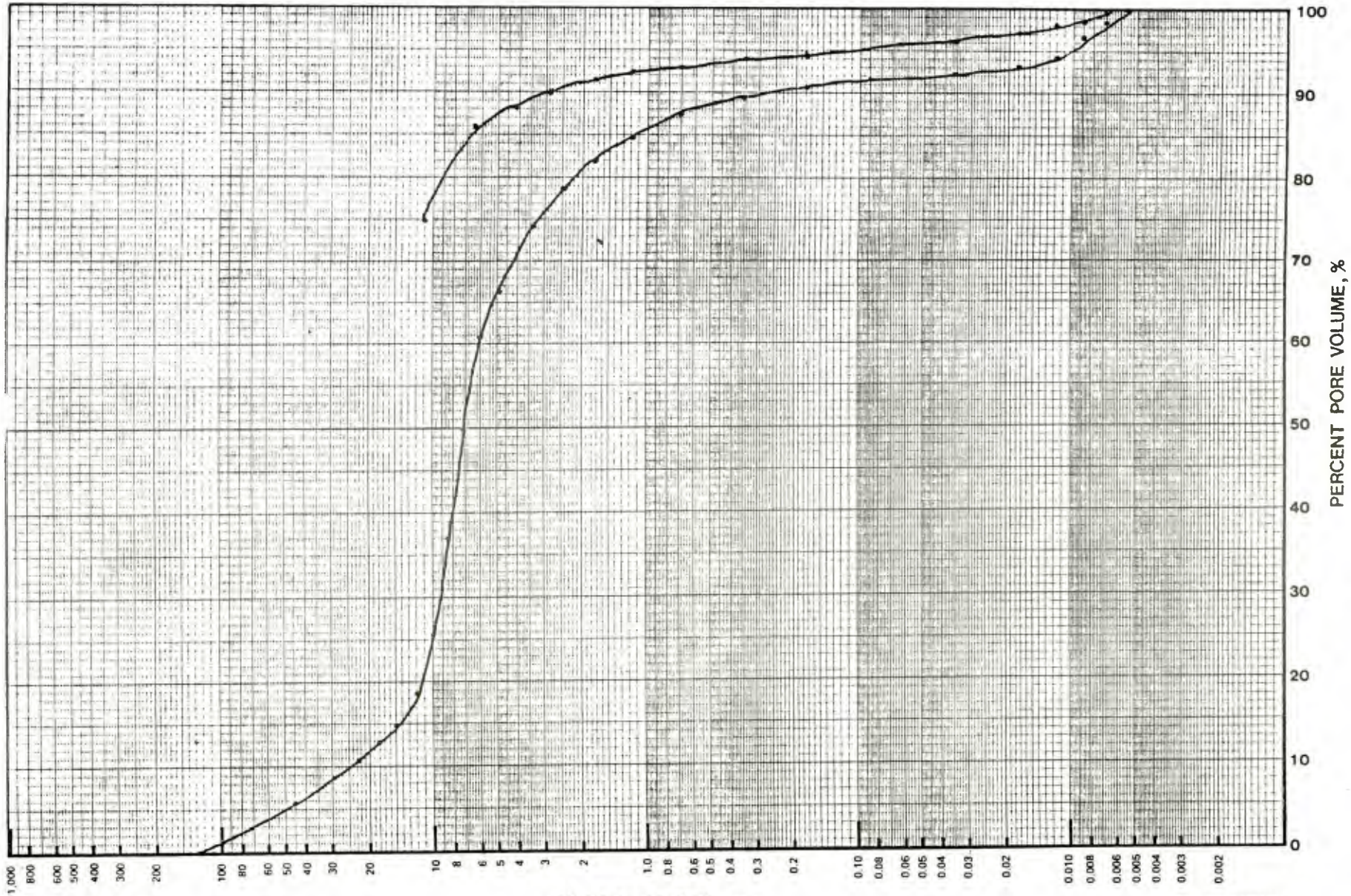
*cc = cm^3

CUMULATIVE PORE SIZE DISTRIBUTION

SAMPLE AIRRADCOM. PICATINNY (NITROCELLULOSE PAPER.) DATE 1/13/81

WEIGHT 0.5252 g PORE VOLUME 0.456 cc/g MEDIAN PORE DIAMETER 7.2 μm BY _____

INTERFACIAL CONTACT ANGLE, θ = 130° MERCURY SURFACE TENSION, γ = 485 dynes/cm



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APPENDIX C

POROSIMETER SUPPLIERS

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Porous Materials, Inc.
Cornell Industry Research Park
Building 4
Ithaca, NY 14850
(607) 257-4267

Quantachrome Corporation
6 Aerial Way
Syosset, NY 11791
(516) 935-2240

Super Pressure, Inc.
(Formerly American Instrument Company)
8030 Georgia Avenue
Silver Springs, MD 20910
(301) 589-1727

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and Development Command
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and Development Command
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  Readiness Command
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ATTN: ATAA-SL
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2185 Fifth Avenue
Ronkonkoma, NY 11779

ARMTEC Defense Products, Inc.
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P.O. Box 848
Coachella, CA 92236

Indiana Army Ammunition Plant
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Charlestown, IN 47111